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Initial Study of Selective Laser Melting of $\text{ZrO}_2/\text{Al}_2\text{O}_3$ Ceramic

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Abstract

Selective Laser Melting (SLM) has been used formation of various metal parts, but the use on ceramic parts is still lacking. Initial studies of SLM of $\text{ZrO}_2/\text{Al}_2\text{O}_3$ ceramic powder system is conducted to observe the processing. Scanning Electron Microscopy and X-Ray Diffraction are used for surface and phase change examination.

Key Words: Laser; Selective Laser Melting; Oxide Ceramic; Process behavior; Parameters.

1. Introduction

Selective Laser Melting (SLM) is an additive manufacturing technology which can build complex shape parts from powder (Chua *et al.* 2010). The SLM process begins with a deposition of a thin layer of powders on a substrate. Then, the powder layer is selectively melted by high energy laser beam to form a melt pool at specific location according to a predetermined pattern. As the laser moves away, the melted place undergoes rapid solidification. Then another layer of powder is placed over the solidified place, and the laser melts it again. As this process proceeds, a 3-D pattern forms. In this way, parts are built layer by layer according to given CAD file data. This process can produce high density structures (Yadroitsev *et al.* 2010, Van *et al.* 2008). Many kinds of metallic powders can be used in SLM technology with good forming quality and excellent mechanical properties, including Ti6Al4V (Lore *et al.* 2010), Inconel 718 (Wang *et al.* 2012), 316L Stainless Steel (Badrossamay *et al.* 2007), Co-Cr alloy (Maria *et al.* 2011) and so on. SLM has its advantages in fabricating net-shaped and high performance composite parts.

Ceramics are inorganic and non-metallic materials with wide range of applications in mechanical and medical fields because of their superior strength, high wear resistance and outstanding chemical property (Massimiliano *et al.* 2004, Isabelle *et al.* 2010). Traditionally ceramic parts are manufactured through power compaction and liquid phase sintering, which usually need post heat treatment for high densification and thus high cost and shrinkage. Rapid manufacturing technology is thus a focus of worldwide research (Filser *et al.* 2003, Bertrand *et al.* 2007) to build high strength and complex shape ceramic structures. Selective Laser Melting makes it possible to manufacture full dense, high strength and complex shape ceramic structures.

$\text{ZrO}_2/\text{Al}_2\text{O}_3$ ceramic system has been intensively studied for dental application using sintering process. With SLM, German researchers Waikes *et al.* (2013) built net-shaped objects of $\text{ZrO}_2/\text{Al}_2\text{O}_3$ ceramic system with almost 100% density and flexural strength of more than 500 MPa. Intensive studies of laser melting of this ceramic system are still lacking.

This paper investigates the SLM process of $\text{ZrO}_2/\text{Al}_2\text{O}_3$ ceramic system in an attempt to reveal the parametric responses of the surface morphology, level and kind of defects as well as the phase change.

2. Experimental procedure

The powder mixture is prepared from two commercial powders: yttria-stabilized zirconia (3YSZ, $\text{ZrO}_2+\text{HfO}_2$ 94wt%, Y_2O_3 6wt%) and high purity alumina ($\alpha\text{-Al}_2\text{O}_3$, 99.8%), at a ratio of 41.5wt% ZrO_2 /58.5wt% Al_2O_3 according to $\text{ZrO}_2\text{-Al}_2\text{O}_3$ phase diagram, as seen in Figure 1. The powder particles of zirconia and alumina are spherical shape with good flow ability, which is quite important for ceramic powder layer deposition. Scanning Electron Microscopy

(SEM) image of the powder mixture is shown in Figure 2. A 6061 aluminum alloy platform is used as the substrate. The phase analysis of the powders before and after the laser melting process is carried out by using X-ray diffraction (XRD).

Figure 1. Phase diagram of alumina and zirconia system

Figure 2. SEM image of zirconia and alumina mixture powder

The powder mixture is layered 100µm thick on to the substrate by a leveling system capable of 20-100µm. An SLM®250 HL system of SLM Solutions GmbH is used to generate laser power of 80~200 W as parameters. The fiber laser beam is 80 µm in diameter with Gaussian beam profile (Laser 1 of the system) and wavelength of $\lambda=1070\text{nm}$. Laser 1 has a capacity of a maximum power of 400 W.

During the process, the laser is directed through an optical window into processing chamber which contains the substrate and the powder supply and leveling system. The chamber is filled with high purity argon to control the oxygen level lower than 0.2%. An optimized scanning strategy of multiple layering in different orientation is used to minimize possible thermal gradient. The scanning speed used are 150mm/s-300mm/s.

3. Results

3.1 Observations of the SLM process

a). The $\text{ZrO}_2/\text{Al}_2\text{O}_3$ powder mixture is white in color structures. After the laser melting processing, the structures built became black in color, probably due to oxygen depletion during the SLM process.

b). Inhomogeneous leveling of powders was observed to occur after a few layers buildup, black streaks are seen as such that in Figure 3. That is probably due to hindrance of leveling from the broken ceramic fragments of previous scanning due to thermal strain. This leads to cracking, delaminating and even warping. Figure 4 shows a sample of defects in this experiment.

Figure 3. The ceramic fragment affect the powder deposition

Figure 4. Defects of ceramic parts after SLM proces

3.2 Phase change

Figure 5 is the X-ray diffraction profile of the ceramic before (a) and after (b) the laser melting process. To start with, the powder mixture is mainly tetragonal zirconia ($t\text{-ZrO}_2$) and $\alpha\text{-Al}_2\text{O}_3$ with a small proportion of monoclinic zirconia ($m\text{-ZrO}_2$). After laser melting, the main phases basically remain, but the monoclinic zirconia phase disappeared owing to the stabilizing effect of Y_2O_3 (conversion from tetragonal phase back to monoclinic does not take place during cooling). Careful examination of the spectrum reveals some degree of peak shifts, which indicate change of crystal lattice parameter. It needs further verification.

Figure 5 XRD of powder materials and SLM parts

Figure 6 is an SEM image of the top surface of an SLM part. The surface is highly dense and smooth, without pores, but with cracks of width in micrometer range cross the laser tracks, due most likely to thermal stress. Further investigation of process parameters should resolve the problem. Also observed on the surface are a small amount of ceramic balls (c.f., Figure 6). This is called the balling effect, a typical defect in SLM (Agarwala *et al.* 1995), caused by poor wet ability of liquid ceramic and inappropriate processing parameters.

Figure 6 SEM of multi layers top surface

4. Summary

ZrO₂/Al₂O₃ ceramic powder system is mixed from yttria-stabilized zirconia and high purity alumina at a ratio of 41.5wt%ZrO₂/58.5wt%Al₂O₃. Selective Laser Melting (SLM) technique has been used to test process 3-D ZrO₂/Al₂O₃ ceramic parts of 20 mm×20 mm×1.3 mm. Color change from white powder to black part is observed, suspected of oxygen depletion. The monoclinic zirconia phase in the starting powders disappeared after the SLM process. Slight dihedral angle shift of the main phases is also observed due to lattice parameter change likely as a result of oxygen depletion. More detailed studies will follow.

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